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(54) Title: ICE-CREAM COATING FATS			
(57) Abstract <p>Novel ice-cream coating fats based on diglycerides comprise at least 30 wt. %, preferably 50-90 wt. %, of diglycerides, which diglycerides have an SU content of 10-25 wt. %, while the total fat composition has a SAFA content of 5-35 wt. % and an N line (unstab.) of N₂₀<35 and N₂₅<10.</p>			

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ICE-CREAM COATING FATS

Ice-cream coating fats known so far are mainly based on triglycerides, in particular triglycerides containing
5 medium-chain fatty acid residues, such as lauric acid residues. Typical examples of such fats are disclosed in, e.g., EP 23,150, US 5,017,392, US 4,560,563, US 4,086,370 and US 3,959,516. However, hydrogenated vegetable non-lauric fats and triglycerides high in polyunsaturated fatty
10 acids are also known as ice-cream coating fats, e.g. from EP 502,697, EP 246,366, EP 23,151 and US 3,333,968.

Hitherto, no composition has been disclosed that is based on diglycerides and is suitable as ice-cream coating fat.

15 According to EP 402,090 oil-in-water emulsions are known, in which the fat phase comprises 10-99 wt.% of a diglyceride mixture having an increasing melting point of at most 20°C, which mixture can also contain some
20 monoglycerides, the total glyceride blend having a melting point of 35°C or below. These emulsions are suitable as cream alternatives and for ice-cream application. For the latter application, however, the emulsion is used for the ice-cream mass and not for the coating of the ice cream.

25 We have studied how to develop ice-cream coating fats that have a low SAFA content (a maximum of 35 wt.%) and an N-line that renders them suitable as ice-cream coating fat and which fat composition would be based on the presence of
30 a minimum amount of diglycerides. The above-mentioned study resulted in novel fat compositions being found that are suitable for ice-cream coatings. These novel fat compositions comprise at least 30 wt.%, preferably 50-90 wt.%, of diglycerides, which diglycerides have an SU
35 content of 10-25 wt.% (S = saturated fatty acid residue; U = unsaturated fatty acid residue), while the fat composition displays a SAFA content of 5-35 wt.% and an N

line (NMR pulse, not stabilized) of $N_{20} < 35$, preferably 1.0-20, more preferably 1.0-5.0; $N_{25} < 10$, preferably < 1.0 .

Although known ice-cream coating fats, such as coconut oil
5 or cocoa butter, have an N_{20} of at least 40, it was found, unexpectedly, that fats with a lower N_{20} can also be applied as ice-cream coating fats; however, the fats should contain enough of the required diglycerides.

10 The above-mentioned finding therefore contradicts the general belief that a high N_{20} is a prerequisite for obtaining high crystallisation rates and acceptable drying times.

15 In particular, the diglyceride part of our novel fat compositions has a U_2 content of 75-90 wt.% and an S_2 content below 5 wt.%.

In a preferred embodiment of our invention the fats display
20 an N_0 of more than 35, in particular $N_0 = 45-80$. It was found that, when $N_0 > 80$, the coating became too brittle, while below $N_0 = 35$ the coating was too soft. The N_{20} controls the oral mouthfeel (waxiness) and meltdown of our ice-cream coating compositions.

25 As coconut oil and cocoa butter have relatively high N_{20} values, whereas our fats have low N_{20} values, the oral meltdown of our fat compositions is much shorter than the meltdown of coconut oil- or cocoa butter-based fat
30 compositions.

Our diglyceride compositions are based on diglycerides derived from fatty acid residues with 12-24 C atoms, preferably 16-22 C atoms (for the saturated fatty acid
35 residues (S)), and for the unsaturated fatty acid residues (U) these acids have at least 16 C atoms, preferably 18 C atoms; in particular, U is oleic acid.

Diglycerides can exist as both the sn-1,2 (sn-2,3) and sn-1,3 isomers. Both isomers can be applied in the present invention. In a preferred embodiment of our invention, however, products enriched in the sn-1,3 isomer are applied. Preferably, a ratio of sn-1,3/sn-1,2 diglyceride isomers > 2.5 is applied to increase the melting profile of the blend. Diglyceride fractions enriched in the sn-1,3 isomer can be prepared by fractionation (in solvent or dry); solvent fractionation using hexane is preferred.

Our novel fat compositions can also contain some triglycerides. Preferred compositions also comprise 10-50 wt.% of a vegetable triglyceride composition, preferably having a total ($U_3 + U_2S$) content of at least 50 wt.%.

Although our triglyceride compositions can be manufactured by blending of their components, either as pure components or as mixtures of components, a preferred way of producing our fats is by performing a glycerolysis of a liquid oil and glycerol. This glycerolysis can be performed by using an enzyme, preferably a 1,3-specific enzyme, or by using a base, such as sodium methanolate.

The reaction conditions for an enzymatic conversion of a liquid oil with glycerol are typically :

weight ratio oil : glycerol 1:10 to 100:1;

reaction times : 6-120 hours;

temperature : 0-40°C

enzymes : selected from *Rhizopus*, *Rhizomucor*,

Pseudomonas, *Candida*, and preferably

Humicola. The enzymes are used in amounts of 100-1000 LU/gm of oil.

The oils that can be applied for the above-mentioned conversion are typically : sunflower oil, high-oleic sunflower oil, safflower oil, high-oleic safflower oil, corn oil, cottonseed oil, rapeseed oil, olive oil and

soybean oil.

After separation from residual glycerol, the crude glycerolysis product is processed to produce the final product by evaporation to remove monoglycerides and optionally fractionation (dry or solvent). This can lead to an optimum ratio of diglycerides to triglycerides in the mixture and this post-treatment can be used to control the N values (in all instances NMR pulse measurements were performed on non-stabilised fats, i.e. after the fats had been stored at 0°C for 90 minutes) or the SAFA content of the product. Sometimes it can be suitable to subject the fats as obtained to a refining treatment (using bleaching earth and steam).

15

A typical ice-cream coating composition comprises the following formulation :

- 35-55 wt.% of a polysaccharide, preferably sugar;
- 20 25-65 wt.% of the fat composition according to the invention;
- 0-20 wt% of cocoa powder, preferably 5-15 wt.%;
- 0-10 wt.% of a milk component, preferably skim-milk powder;
- 25 0-2 wt.% of an emulsifier, preferably lecithin.

Part of our invention are also ice creams coated with the above-mentioned ice-cream coating compositions.

EXAMPLE 11.1 Glycerolysis of high-oleic sunflower oil

5 High-oleic sunflower oil (having the composition given below) was reacted with glycerol in a ratio of 5 parts of HOSF oil to 1 part of glycerol by weight, in the presence of *Humicola* lipase (1000 LU/gm of oil). The reaction time was 24 hours and the temperature was 40°C. After separation
 10 from glycerol, monoglycerides were removed by evaporation at 240°C and 1 mbar pressure. The resulting product was fractionated in hexane at -10°C; the product obtained was refined by a treatment with bleaching earth and steaming at 190°C for 4 hours to give a diglyceride-rich fraction of
 15 the following composition :

	<u>C_{16:0}</u>	<u>C_{18:0}</u>	<u>C_{18:1}</u>	<u>C_{18:2}</u>	<u>C₂₀</u>	<u>% SAFA</u>
Product (= Fat A)	3.9	5.7	86.2	1.5	2.6	12.2
HOSF (= Fat B)	4.0	4.7	84.3	4.9	2.1	10.8

20

1.2 Preparation of another diglyceride-product (= fat E)

Two diglyceride-rich fat products (an olein and a stearin) were prepared from the glycerolysis of HOSF, followed by
 25 separation techniques:

- a) 100:20:0.5 by weight of HOSF, glycerol, Lipolase 100 L enzyme (ex-Nove Nordisk) respectively, were stirred at 40°C for 24 hours.
- 30 b) Excess glycerol was decanted off, and mono-glycerides/FFA were removed from the crude reaction product (22.1 wt% DG, 3.8 wt% MG) in a falling film evaporator (260°C, 0.3 mm Hg abs.).
- 35 c) After refining, the diglyceride rich product was fractionated from hexane (2:1 hexane oil to oil by

weight at -12°C), collecting the olein (16.9 wt% DG, yield 90 wt%) and the stearin (63 wt% DG, 7.8 wt% MG, yield 10 wt%).

- 5 d) The olein product had a composition of:
16.9 wt% DG, 0.8 wt% MG
DG part 0.0% SS type, 15.1% SU type, 84.8% UU type.
- e) The diglyceride content of the olein product was
10 increased by a two-stage silica treatment:
i) Diglycerides and monoglycerides were absorbed
onto silica, using hexane as a solvent (in the
proportion 2:1:1 hexane, oil, silica by weight). The
silica complex was washed with hexane (2.6:1 hexane to
15 oil by weight) and the wash discarded.
ii) The silica complex was washed with 88 wt% hexane/
12 wt% acetone (3:2:1 wash to oil by weight), and the
diglyceride-rich wash collected. A diglyceride-rich
fat was formed by evaporation of the hexane/acetone
20 solvent.
- f) The diglyceride enriched olein had a composition of:
51.5 wt% DG, 0.1 wt% MG,
DG part 0.0% SS type, 16.5 % SU type, 83.5% UU type.
25
- g) Excess monoglycerides were removed from the stearin
product via a silica treatment with hexane/acetone (88
wt% hexane, 12 wt% acetone) as the solvent (5:1:1.21
solvent, oil, silica by weight) washed with 3 parts
30 solvent to 1 part oil (by weight). The stearin product
was recovered from the wash solvent by evaporation.
- h) The silica treated stearin had a composition of:
69.6 wt% DG, 0.3 wt% MG
35 DG part 0.6% SS type, 25.1% SU type, 74.4% UU type.

A diglyceride-rich fat blend was prepared by blending the

silica treated stearin and diglyceride enriched olein in the ratio 30:70 by weight. The blend was bleached and deodorised.

- 5 The refined blend contained: 55.5 wt DG, 0.1 wt% MG.
The DG composition was 0.4 % SS, 21.1 % SU, 78.5 % UU.

The FAME profile of the total blend was (wt%):

14:0	16:0	16:1	18:0	18:1	18:2	18:3	20	22	24
0.0	4.0	0.1	4.5	84.9	4.6	0.0	0.6	1.1	0.2

- 10 giving a total SAFA level of 10.1 wt%.

The triglyceride part contained 20 wt% of SOO and 65.9 wt% of OOO.

- 15 1.3 The glyceride compositions of the refined fat A, of the starting oil (= Fat B), of a chemically made product (Fat C), of coconut oil (Fat D) and of the second enzymically made fat (= E) were as follows :

20	(Wt.%)	<u>Triglyceride</u>	<u>Diglyceride</u>	<u>Monoglyceride</u>
	Fat		(1,3 + 1,2)	
	A	14.2	80.3	5.5
	B	98.0	2.0	-
	C	-	98	2
25	D	98.0	2	-
	E	44.4	55.5	0.1

- The melting profile of the refined fat composition was measured. Fat C is a product that is made using a chemical
30 conversion with a base; Fat D is coconut oil.

		<u>Temperature (°C)</u>		
	Solids	0	20	25
	Fat			
35	A	42.0	2.7	0.1
	B	4.1		
	C	63.5	18.8	10.7

D	89.0	37.6	0
E	34.4	0.2	0

2. Ice-cream coatings

2.1 Ice-cream coatings were prepared, using the following

5 recipe :

	<u>Recipe :</u>	<u>wt.%</u>
	sugar	42.7
	fat	38.1
	cocoa powder D-11-MC	12.5
10	SMP	6.2
	lecithin	0.5

The fats applied were : cocoa butter (= CB), coconut oil (CN) and the refined enzymic conversion product of Example 15 1.1 and 1.2.

2.2 Ice creams were dipped in a melt of the above-mentioned ice-cream coating compositions.

20 The following results were obtained :

	<u>Fat</u>	<u>Coating T</u> (°C)	<u>Dripping time</u> (sec)	<u>Drying time</u> (sec)	<u>wt.%</u> of coating
25	CB	40.5	17	122	36
	Enz. conv. product A	41.0	25	99	26
	CN	38.5	22	73	36
30	Enz. conv. product E	40	23	97	32

The oral properties of the products were similar (all 35 good-tasting). However the products according to the invention resulted in coatings that were softer and less brittle, which had quicker and smoother meltdown than CB-based coatings.

CLAIMS

1. Fat composition suitable for ice-cream coatings comprising at least 30 wt.%, preferably 50-90 wt.%, of diglycerides, which diglycerides have an SU content of 10-25 wt.% (S = saturated fatty acid residue; U = unsaturated fatty acid residue), while the fat composition displays a SAFA content of 5-35 wt.% and an N line (NMR pulse, not stabilized) of $N_{20} < 35$, preferably $N_{20} = 1.0-20$, more preferably 1.0-5.0, $N_{25} < 10$, preferably < 1.0.
2. Fat composition according to Claim 1, wherein the diglyceride part has a U_2 content of 75-90 wt.% and an S_2 content below 5 wt.%.
3. Fat composition according to Claims 1-2, wherein the fat compositions displays an $N_0 > 35$, preferably 45-80.
4. Fat composition according to Claims 1-3, wherein S has 12-24 C atoms, preferably 16-22 C atoms, and U has at least 16 C atoms, preferably 18 C atoms.
5. Fat composition according to Claims 1-4, wherein the composition also comprises 10-50 wt.% of a vegetable triglyceride composition, preferably having a total ($U_3 + SU_2$) content of at least 50 wt.%.
6. Ice-cream coating composition comprising :
 - 35-55 wt.% of a polysaccharide, preferably sugar;
 - 25-65 wt.% of the fat composition according to Claims 1-5;
 - 0-20 wt% of cocoa powder;
 - 0-10 wt.% of a milk component, preferably skim-milk powder;
 - 0-2 wt.% of an emulsifier, preferably lecithin.

7. Coated ice creams wherein the ice-cream coating consists of the ice-cream coating according to Claim 6.

INTERNATIONAL SEARCH REPORT

Internati Application No
PCT/EP 95/01572

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A23G9/02 A23D9/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A23G A23D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP-A-0 477 935 (KAO CORPORATION) 1 April 1992 see page 2 - page 4; claims 1,3 ---	1-4,6,7
Y	EP-A-0 402 090 (KAO CORPORATION) 12 December 1990 cited in the application see page 3, line 1-16; claims 1-3 see page 4, line 1-49 ---	1-4,6,7
Y	EP-A-0 023 151 (UNILEVER LTD) 28 January 1981 cited in the application see claims 1,4; example 2 --- -/--	1-4,6,7

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	PATENT ABSTRACTS OF JAPAN vol. 017 no. 693 (C-1144) & JP,A,05 236919 (KAO CORP.) 17 September 1993, see abstract ----	1
A	EP-A-0 483 414 (UNILEVER NV) 6 May 1992 see claims 1-6 ----	1,3,6,7
A	US-A-4 379 176 (THE PILLSBURY COMPANY) 5 April 1983 see column 3; claim 1 -----	5,6

INTERNATIONAL SEARCH REPORT

Information on patent family members

Internat Application No
PCT/EP 95/01572

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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